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SPECTRAL STUDIES OF
SOLID PROPELLANT COMBUSTION
III. EMISSION AND ABSORPTION RESULTS
FOR HMX2 PROPELLANT

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13. ABSTRACT (Maximum 200 words) Optical absorption and emission spectroscopy has been applied to HMX2 (a nitramine propellant exhibiting a "dark zone") burning over a pressure range of 1.2 to 1.8 MPa nitrogen. Emission signals from CN have been observed and profiled, as well as absorption signals from NO. Large broadband absorptions occurred in the wavelength range appropriate for NO absorption. There was significant NO present in the dark zone as evidenced by 100% absorption occurring in the (0,0) vibrational band of the $A^2\Sigma - X^2\Pi$ transition.				
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I. INTRODUCTION

During the last several years we have been developing an experimental facility to study solid propellant combustion under moderate pressure conditions (0.1 to 2.0 MPa). Three techniques have been used to obtain diagnostic information: video recordings, emission spectroscopy and absorption spectroscopy. The wavelength range for the spectroscopic studies has been confined to the near ultraviolet region. Concentrations of diatomic radical species as well as temperatures have been obtained for two propellants, M-30 and HMX1.^{1,2} The results reported here are an extension of this previous work but using a different propellant.

The propellant under study in the present investigation is HMX2. Low pressure characteristics of HMX2 are quite different from either HMX1 or M-30 as can be seen from the information in Table 1. Propellants similar to HMX2 will find application for rockets where slow burning rates and low flame temperatures are desirable and for tank guns where low vulnerability is extremely important.

Of the three propellants thus far studied, HMX2 gave the poorest spectroscopic signals. This is not too surprising as the equilibrium flame temperature is significantly lower for this propellant and the concentration of diatomic combustion transient species is a strong function of temperature. On the other hand this propellant burns like a two stage flame where there is a sizable dark zone between the burning surface of the propellant and the luminous flame. This feature, while decreasing the signal intensity, should provide for better spatial resolution through the reaction zone. However, the poor stability of combustion undermined any gains that might be realized from this effect. The only spectroscopic results obtained were emissions signals for CN and absorption signals for NO.

Table 1. Propellant Characteristics

Propellant	Main Ingredients Wt. %	Equil. Flame Temperature (K)	Burn Rate at 1.5 MPa (mm/s)	Darkzone Length at 1.5MPa (mm)
HMX2	80% HMX 20% PE Binder	2045 @ 1.5 MPa	0.7	3
HMX1	73% HMX 10% PE Binder 17% TMETN	2617 @ 1.5 MPa	1.1	Not Observable < 0.1 mm
M-30	48% Nitroguanidine 28% Nitrocellulose 23% Nitroglycerin	2423 @ 1.0 MPa	3.1	Not Observable < 0.1 mm

HMX - cyclotetramethylene tetranitramine, PE - polyester binder based on polydiethylene glycol adipate
TMETN - trimethylethane trinitrate

II. EXPERIMENTAL

The windowed pressure chamber, optical paths and motorized feed have been discussed in detail previously^{1,2} and thus only a brief description will be given here. A top view of the experiment is shown on Fig. 1. The chamber has five optical ports: one at the top and two sets of opposed ports. A critical orifice allows for a pressuring gas flow while maintaining constant pressure. This flow is directed around the propellant sample to approximate a shroud gas and the magnitude is about four times the propellant gasification rate. Propellant samples are mounted on a cylindrical rod which can be raised remotely by a motorized feed-through situated in the chamber base. This drive can raise the propellant at a predetermined rate during the burning process. The propellant samples approximated cylinders of 0.65 cm diameter and the most common length used was 2.0 cm.

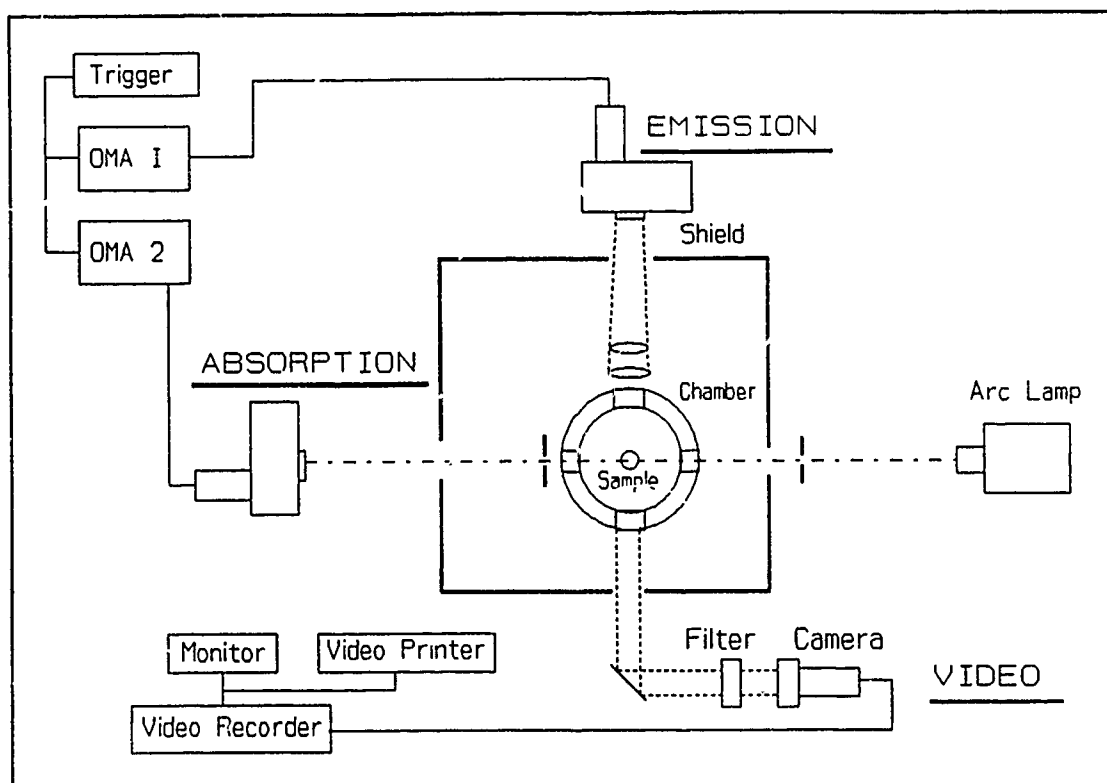


Figure 1. Sketch of the experimental apparatus used to perform optical studies of propellant combustion.

Video recording was accomplished with a super VHS system composed of a JVC BY-10U CCD color video camera and a Panasonic AG-1830 VGS video cassette recorder. Magnification factors (horizontal and vertical) of the system were determined from static measurements and used to ascertain: burn rates and dark zone distances from real time playbacks. Real-time video provided information for use in determining when to trigger other equipment; it also reveals whether the propellant sample is burning in a cigarette fashion with the burning surface parallel to the absorption light beam.

The emission was collected and focussed on the entrance slit of a monochromator with two convex lenses. A 1/4 m Jarrell-Ash monochromator with a 1200 groove/mm grating was used to disperse the light emission signal onto an intensified photodiode array detector system (EG&G Princeton Applied Research Model 1455B detector, Model 1462 detector controller, and a Model

1460 console). The emission signal collection optics were positioned such that the light was collected from the same region that was being probed by optical absorption. Data gathering for emission and absorption was coordinated by using a single external pulse to initiate the scanning/memory activity of the emission detector which in turn activated the the absorption detector with a synchronization pulse being sent for each scan. This feature insured a time differential of no more than a few milliseconds between the two detector systems.

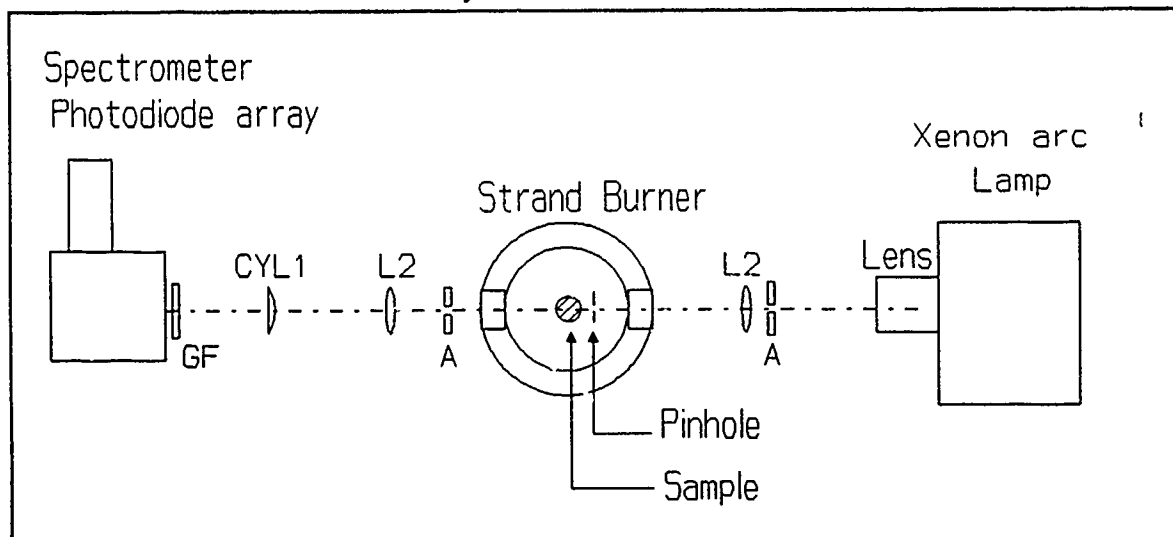


Figure 2. Top view of the optical path for absorption measurements.

Spatial- and time-resolved absorption measurements were made by passing a light beam from an arc lamp through the windowed chamber as shown in Fig. 1. A more detailed top view of the absorption path for the arc lamp is shown on Fig. 2. A 500 watt cw xenon arc with close electrode spacing is the broad-band light source. This light is focussed with a convex lens attached to the arc lamp housing. Further focussing and recollimating is accomplished with two convex lenses (L2) each of 20 cm focal length. Two apertures (A) act to confine the beam. The aperture between the sample and the detector minimizes collection of emission light. Spatial resolution is set by the 0.1 mm pinhole in close proximity to the sample. The final optic is a cylindrical lens (CYL1) which focusses the light to a line on the entrance slit of a 0.32 m JY monochromator Model HR-320 with a 2400 grooves/mm grating. An intensified photodiode array detector system (EG&G Princeton Applied Research Model 1420 detector, Model 1463 detector controller and Model 1460 console) captures a spectral range of 6 or 12 nm depending on whether the monochromator is operated first or second order. For second order operation, a glass filter (GF) is required to remove first order light. The spectral data is digitized and stored in the console for further analysis on a PC.

In the absorption measurements the wavelength resolved intensity of the light source is the primary measurement. This measurement is taken for conditions where the absorber of interest is absent (i.e., the incident intensity I_0) and where the absorber is present (i.e., the transmitted intensity I). The absorption is typically represented as the ratio (I/I_0). In this study I_0 was measured after the chamber was closed and pressurized; but prior to combustion of the propellant. In some cases, I_0 measurements were taken immediately after the completion of burning but these were generally of poorer quality due to an accumulation of smoke that had settled down into parts of the absorption path. During the propellant burn the history of the transmitted beam was measured by collecting, typically, six 20 ms scans which were stored in memory and this process was repeated for 80 memories. Total sampling times ranged from about 10 to 20 seconds with a time resolution of 0.12 s.

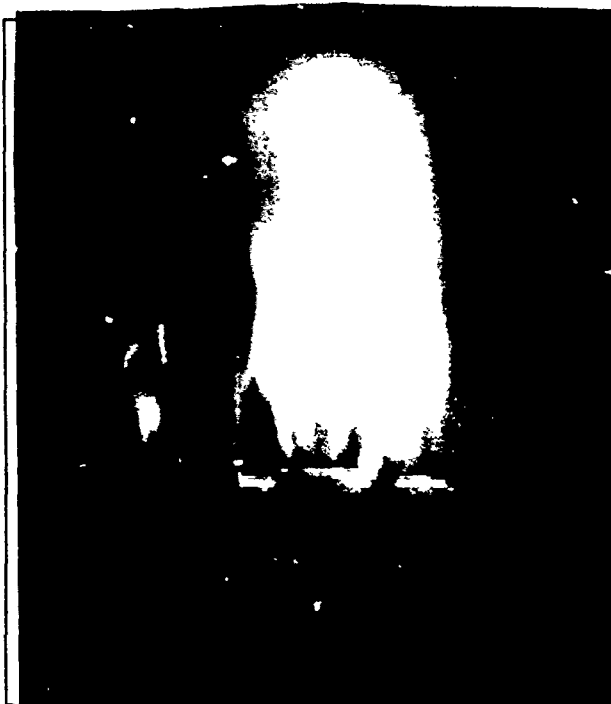


Figure 3. Video frame of HMX2 burning at 1.6 MPa.

A typical data-gathering sequence involved the following steps: (1) loading and pressurizing the windowed chamber, (2) checking the absorption optical path and measuring I_0 , (3) raising the propellant into the absorption beam path with the motorized feed, (4) starting the video recording, (5) switching the current to the ignition wire, (6) triggering the optical detectors at an appropriate time determined from the video monitor, and (7) triggering the motorized feed to increase the data acquisition time when desired.

III. RESULTS

Some of the characteristics of the burning of HMX2 propellant can be conveyed by the photograph shown as Fig. 3; it is estimated that visible light output as detected by the video camera is two orders of magnitude less than that which would be produced from HMX1 burning at the same pressure of nitrogen. Here the dark zone between the burning surface of the propellant and the luminous flame is apparent. Additionally, wisps of luminosity projecting toward the propellant surface can be seen on the photograph. This is one of the better video frames for showing the presence of the dark zone; many other frames in the sequence demonstrate strong attachment of the flame to the surface. The burning surface appears to be a liquid layer where droplets of molten propellant appear from time to time on the burning surface. These droplets vary in shade, but tend to be dark, probably due to the pyrolysis of the HMX and/or binder. Often the flame attaches to these droplets, burning directly on the surface. This burning is accompanied by a greater emission of light than that above the dark zone. Occasionally, the burning of the droplets produces a solid ash that glows before being consumed. Over the pressure range studied there appeared to be no correlation between the relative flame attachment time and pressure. Burning HMX2 in nitrogen at these moderate pressures produces a large amount of smoke. This smoke can interfere with the spectroscopic measurements and in order to minimize the effect the propellant burns were of short duration so that smoke buildup would be minimal. Moreover, the majority of the path of the absorption light beam within the chamber was enclosed by tubes with a small hole in the ends (3mm). Only the region near the propellant sample was open for smoke and this area is purged by a positive flow of nitrogen shroud gas. Generally, the experimental data is collected temporally and then converted to a distance from the propellant surface. This conversion factor, which is pressure dependent, is the burn rate which can be determined from the video recording. The burn rate for HMX2 as a function of pressure is shown on Fig. 4. Burn rate data for HMX2 as measured by Edwards³ and Kubota⁴ are also included on the graph. The agreement with Edwards is excellent; there is a small offset with the Kubota data, however, the slopes are the same. (The difference in the Kubota propellant is that the binder is polyurethane). Over the pressure range of 1.2 to 1.8 MPa a linear least squares fit to the data gives a burn rate of $(0.019 + 0.475P)$ mm/s for HMX2 in nitrogen where the pressure is expressed as MPa.

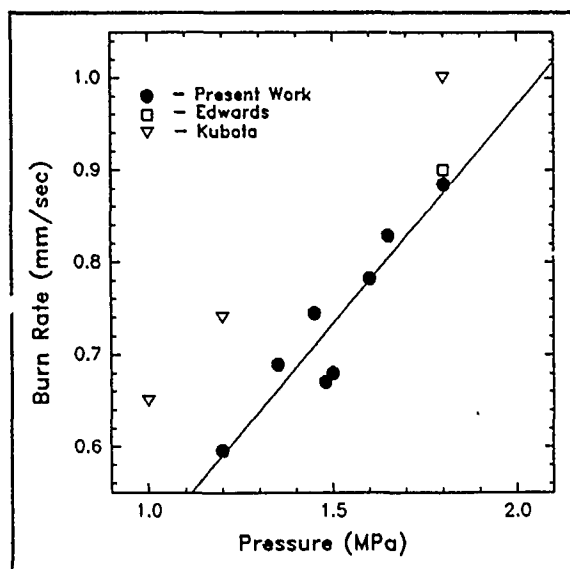


Figure 4. The burn rate of HMX2 as a function of nitrogen pressure. The solid line is a least squares fit to the data.

A search was made for NH, OH and CN emission signals in HMX2 burning over the range of 1.2 to 1.8 MPa nitrogen. No emission signals were observed for OH and only weak signals could be obtained for NH emission. The strongest emission signals came from CN and a typical emission spectrum taken from burning HMX2 is shown in Fig. 6. The (0,0) vibrational bandhead for the $B^2\Sigma - X^2\Sigma$ transition of CN is readily observed. Many of these CN spectra are taken during the time of a propellant burn and the integrated emission intensities taken from many (80) CN spectra like Fig. 6 as a function of distance from the propellant surface are illustrated in Fig. 7. The burning propellant surface has been located on the graph by noting the first transmission of light from the absorption beam which has been positioned in the focus of the emission collection optics. There is essentially no observed CN emission for the first several millimeters from the surface (dark zone region) then the CN emission appears abruptly, fluctuating over the next 10 millimeters. In general, CN is a transient combustion species which only appears over a short distance or region which is commonly referred to as the reaction zone. For flames operating at atmospheric pressure the length of the reaction zone is about 1 mm. Hence shorter reactions zones would seem appropriate for these propellant burning conditions. Consequently, a degradation in spatial resolution is probably occurring for these CN emission intensity measurements. Video recordings show definite spatial and light intensity fluctuations in the combustion process and these fluctuations can degrade the spatial resolution of a technique defined by one optic.

At low pressure the dark zone is readily observable in HMX2. The height of the dark zone above the burning surface decreases with increasing pressure and this variation is plotted in Fig. 5. Other data shown for comparison are those of Kubota for the propellant with a polyurethane binder. Within the estimated error of the measurements, the agreement is quite good. Fluctuations and flame attachment accounted for the scatter. The quality of this type of data may be improved by using larger size propellant samples since, when operating in a region close to the lower limit of self sustained burning, geometric factors could become important. Extending the pressure range to higher pressures may be beneficial in helping to minimize fluctuations in the combustion process. However, there will be a point where the extent of the dark zone becomes too small to be adequately measured.

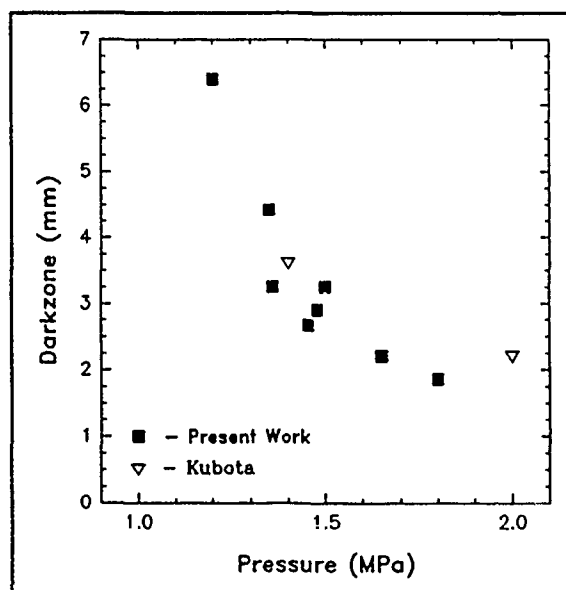


Figure 5. The length of the darkzone above the propellant surface as a function of nitrogen pressure for HMX2 propellant.

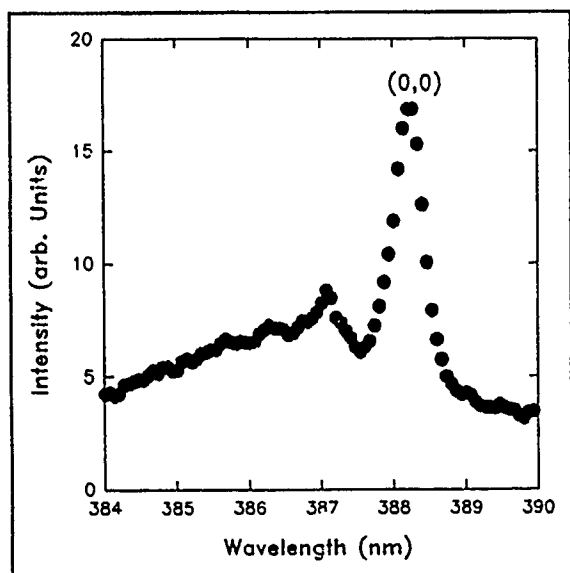


Figure 6. CN emission spectrum obtained in HMX2 burning in 1.8MPa nitrogen.

OH and CN absorptions were not detected in the gas combustion region of burning HMX2. NO absorptions were readily detected but suffered from large attenuations from at least one other source besides NO. An NO absorption spectrum is shown on Fig. 8. The amount of absorption is represented by the transmittance (I/I_0) plotted as a function of wavelength over the range which includes the (0,1) and (0,2) vibrational bandheads of the $A^2\Sigma - X^2\Pi$ transition in NO. The (0,0) bandhead was not used since 100% absorption occurred there. In addition to the features of the spectrum which we attribute to the (0,1) and (0,2) bandheads of NO there is a tremendous broadband attenuation (maximum transmission is about 8%) occurring over this wavelength region. The cause of this attenuation has not been experimentally determined but several possible mechanisms come to mind. First, the propellant

produces particulates in the gas phase combustion which scatter substantial amounts of light; supporting evidence for this is the powder-like residue left in the sample chamber after burning the HMX2 propellant. While this may contribute to the attenuation of the arc lamp beam, it is not thought to be the dominant process because at wavelengths appropriate for OH absorption there is over 50% transmission of the light signal. Another possible source of attenuation is broadband absorption by undetermined molecular species. It has been found that either HMX or RDX in solution exhibits a large absorption envelope over the region from 200 to 300 nm.⁵ Parr and Parr⁶ have also observed large broadband absorption over this region which they suggest is from nitramine absorption. Since this absorption is so large temperatures and concentrations extracted from these spectra would be questionable. Instead the per cent NO absorption was plotted (neglecting the broadband absorption contribution) as a function of distance from the propellant surface for both the (0,1) and (0,2) vibrational bands. This plot is shown in Fig. 9. There is about 50% absorption for the (0,1) band, out to about 2mm from the propellant surface at which point the absorption starts to decrease. No data at larger distances were obtained because the propellant length was insufficient when using the feed mechanism. The feed mechanism was used in this run to increase data acquisition time over a smaller spatial extent. In other runs (not shown) the absorption signal drops precipitously in the region from 2 to 3 mm from the propellant surface. From the video results displayed on Fig. 3 the dark zone length is about 2 mm at 1.8 MPa. It is thought that the conversion of NO_2 to NO produces a large fraction of NO which reacts slowly in the dark zone until the onset of reactions which convert NO to N_2 . The absorption profiles for NO

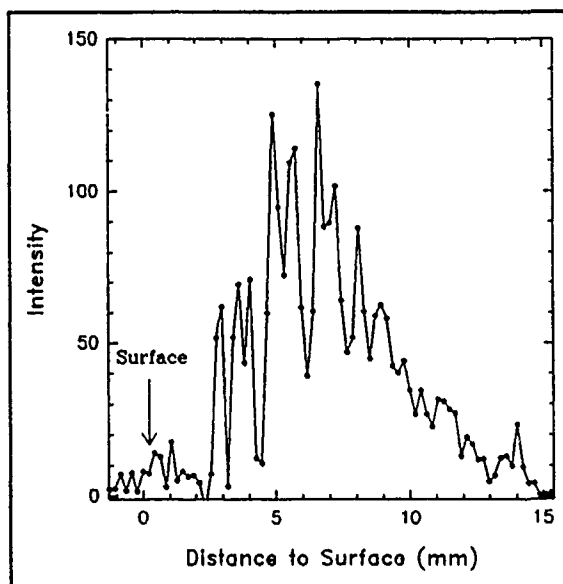


Figure 7. CN emission intensity as a function of distance for HMX2 burning in 1.8 MPa nitrogen.

are consistent with this type of mechanism. The $(0,2)/(0,1)$ ratio for per cent NO absorption yields a temperature of about 1300 K assuming a Boltzmann distribution of states. This temperature value is in excellent agreement with the dark zone temperatures Kubota⁴ obtained using micro-thermocouples.

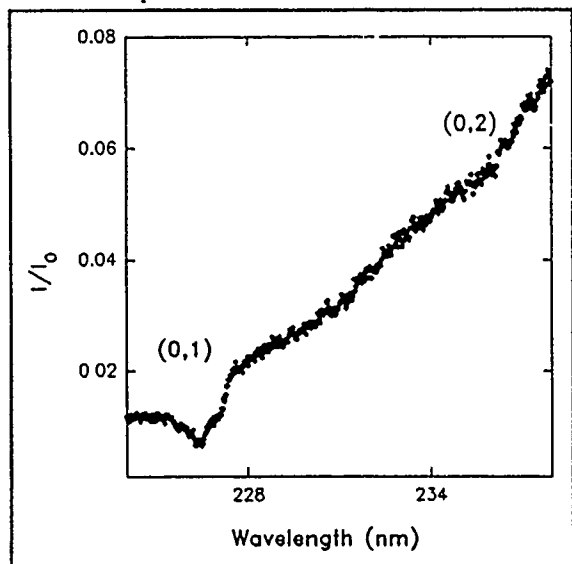


Figure 8. NO absorption spectrum obtained in HMX2 burning in 1.8 MPa nitrogen.

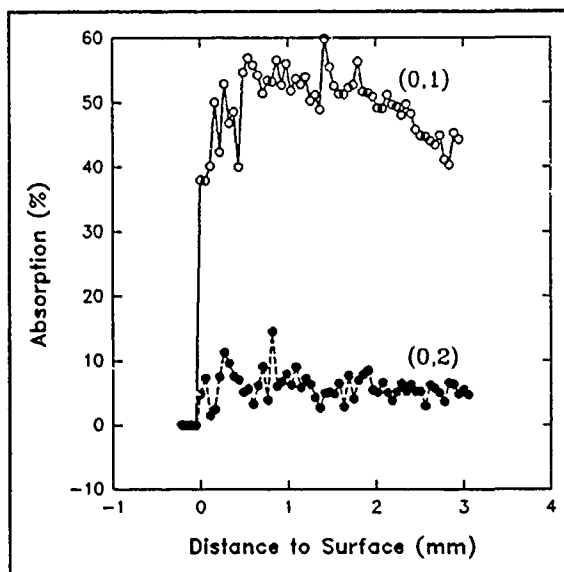


Figure 9. NO absorption [(0,1) and (0,2) bands] as a function of distance for HMX2 at 1.8 MPa.

IV. CONCLUSIONS

It is much more difficult to extract diagnostic information from HMX2 than from HMX1 using uv-visible absorption or emission spectroscopy. The large difference in the adiabatic flame temperatures is responsible for much of the difficulty (i.e. substantially lower concentrations of transient combustion species produced in HMX2). Moreover, a large broadband absorption was present throughout the NO absorption wavelength region. Nonetheless, the results of this paper confirm earlier measurements of large quantities of NO in the dark zone, are consistent with generally accepted chemical mechanism for the existence of a propellant dark zone, and illustrate experimental difficulties that can be encountered when applying spectroscopic techniques to a propellant that produces a dark zone.

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